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Membrane Emulsification for the Continuous Production of Cellulose Beads



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Cellulose Dissolution

An estimated 1.5×10^{12} tons of cellulose is generated every year making it the most abundant biopolymer on the planet.¹ However, the wide scale sustainable use of this almost inexhaustible raw material is hindered by its low solubility by virtue of an extensive hydrogen bonding network (Figure 1, A).

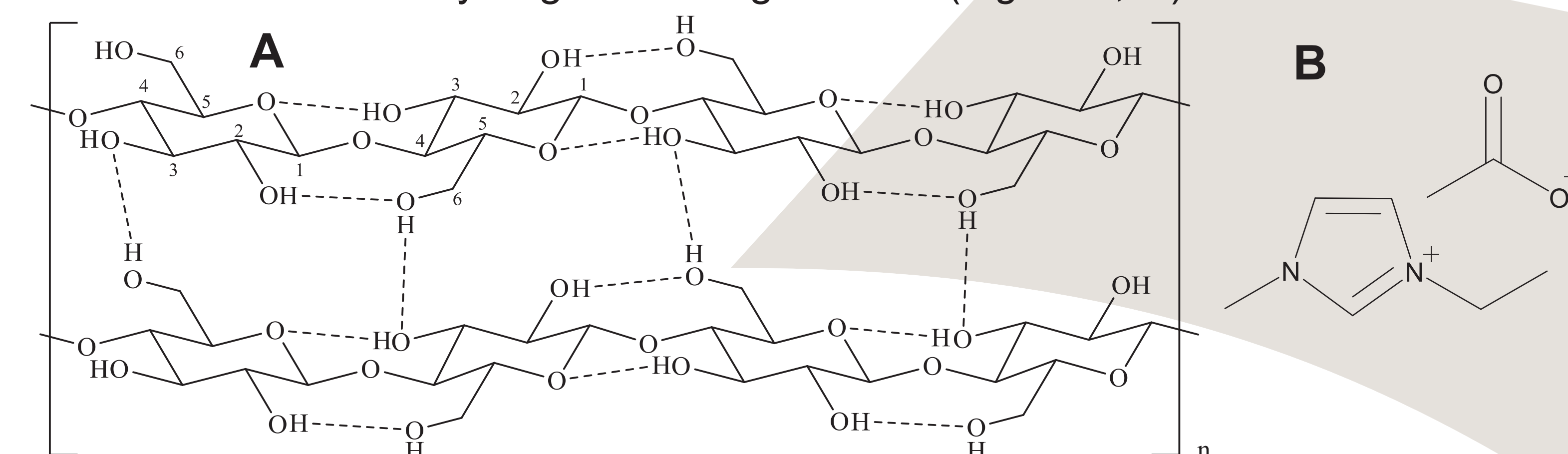
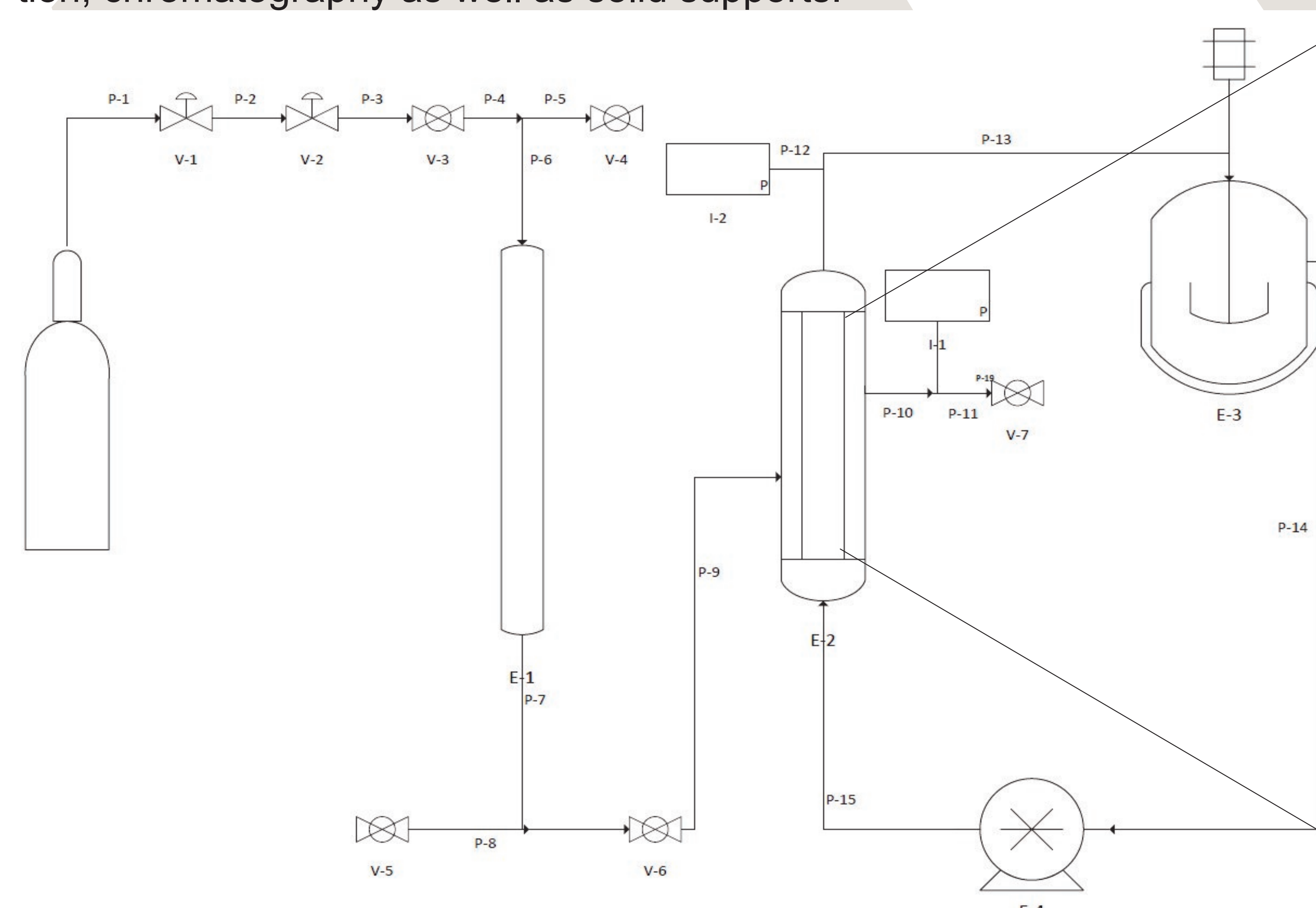


Figure 1: **A** = Diagram of the inter and intra-chain bonding of cellulose.^{2,3}

B = the ionic liquid 1-ethyl-3-methylimidazolium acetate

Ionic liquids (ILs) — molten salts with a melting point $<100^\circ\text{C}$ —can solvate cellulose opening up a potential processing avenue especially when used with a co-solvent such as DMSO (i.e. reduced viscosity, faster dissolution, less ionic liquid).^{4, 5}

This research shows the use of such a solvent system (EMIMAc, DMSO, Figure 1 B) to process cellulose solutions via a membrane emulsification technique into sustainable and industrially important cellulose beads often used in water filtration, chromatography as well as solid supports.



Membrane Emulsification

Emulsions, a mixture of two immiscible liquids, are usually formed via high shear force processes but there is a drive towards lower energy and less harsh membrane emulsification techniques which also provide greater control over dispersity and droplet size (Figure 2).

Water in Oil emulsions (W/O) were used as a precursor to cellulose bead products. Specifically, solvated cellulose solutions were dispersed in a continuous sunflower oil phase then subject to an anti-solvent producing solid cellulose beads (Figure 3).

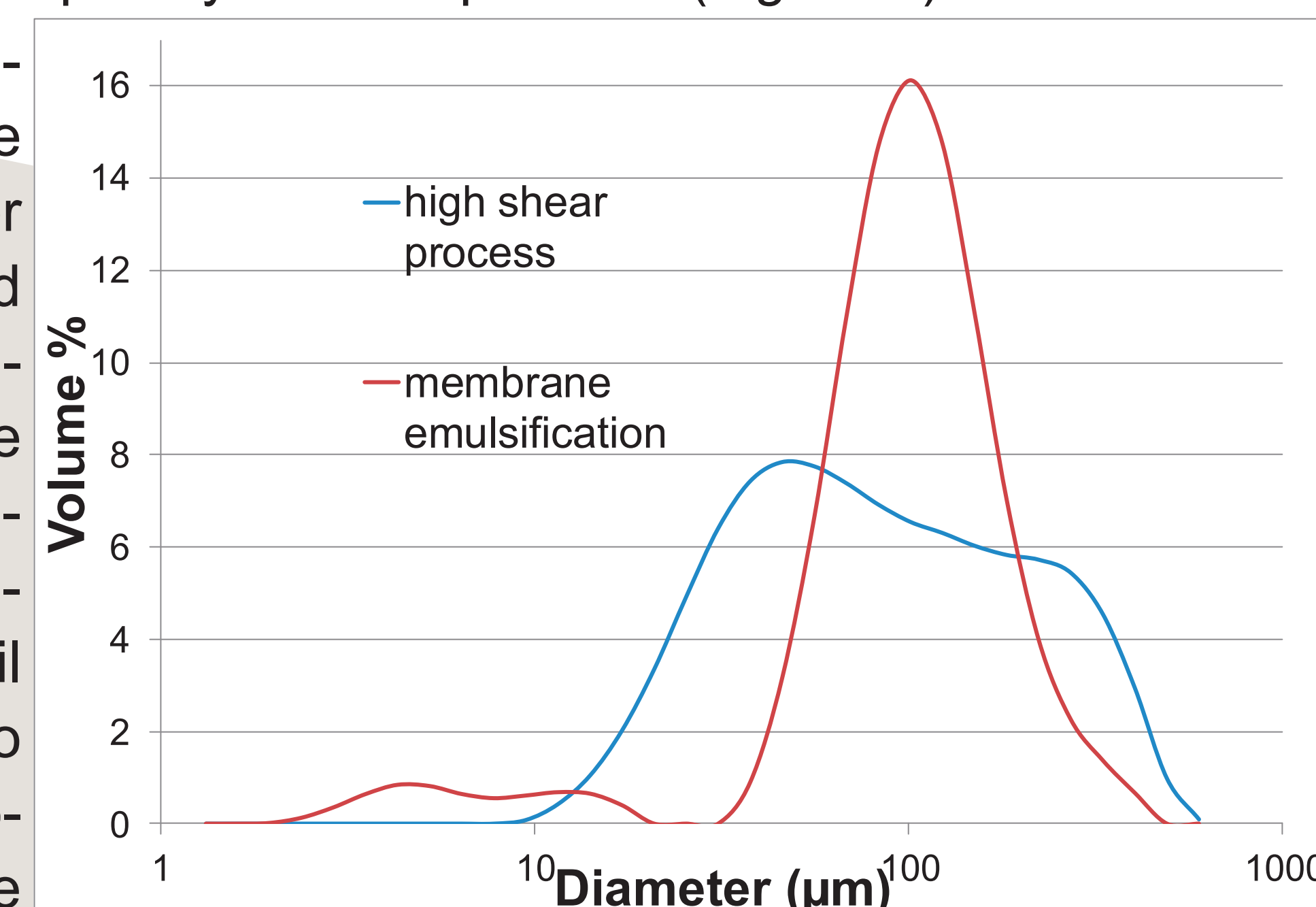


Figure 2: A comparison between the dispersity and average size of cellulose beads made via two techniques.

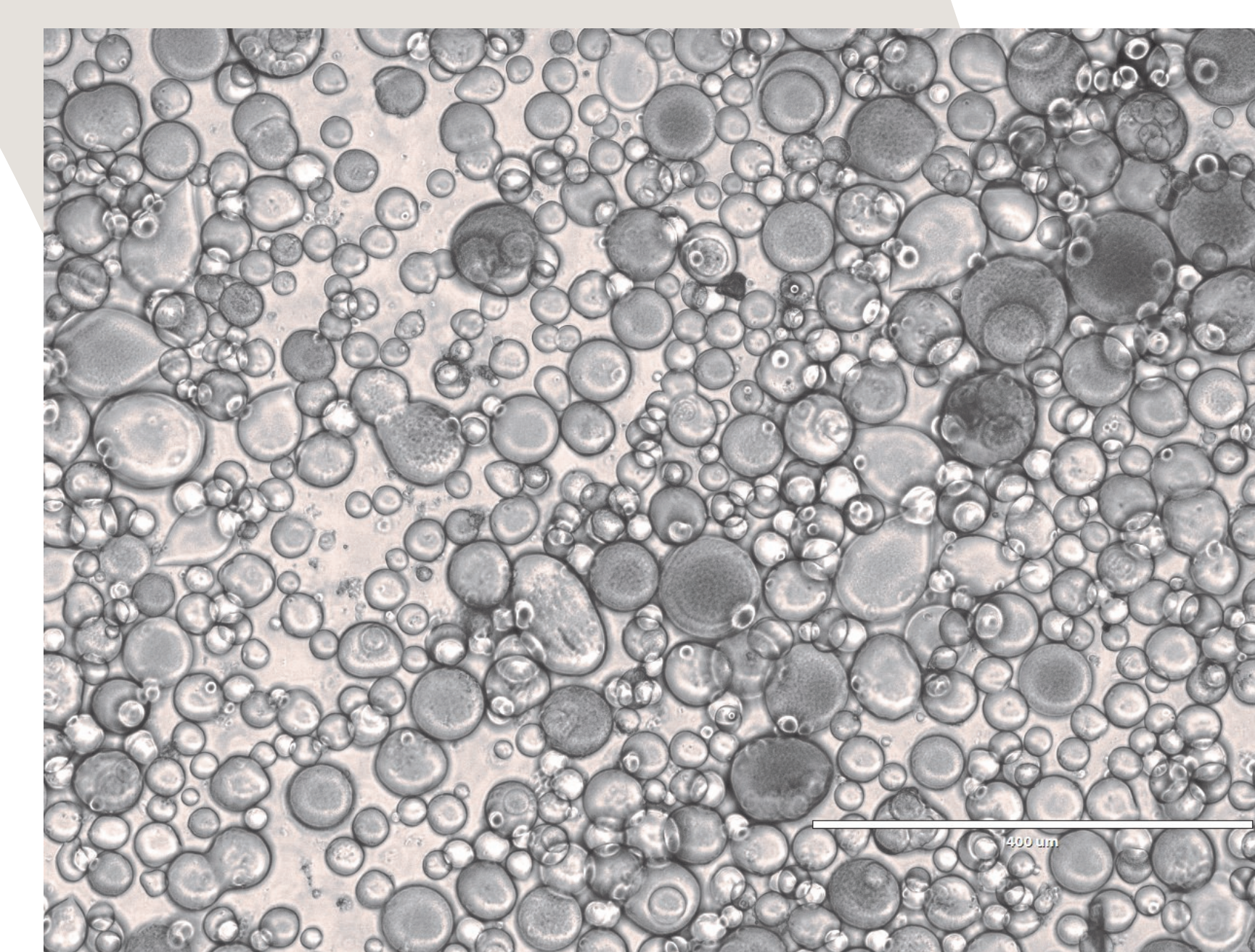
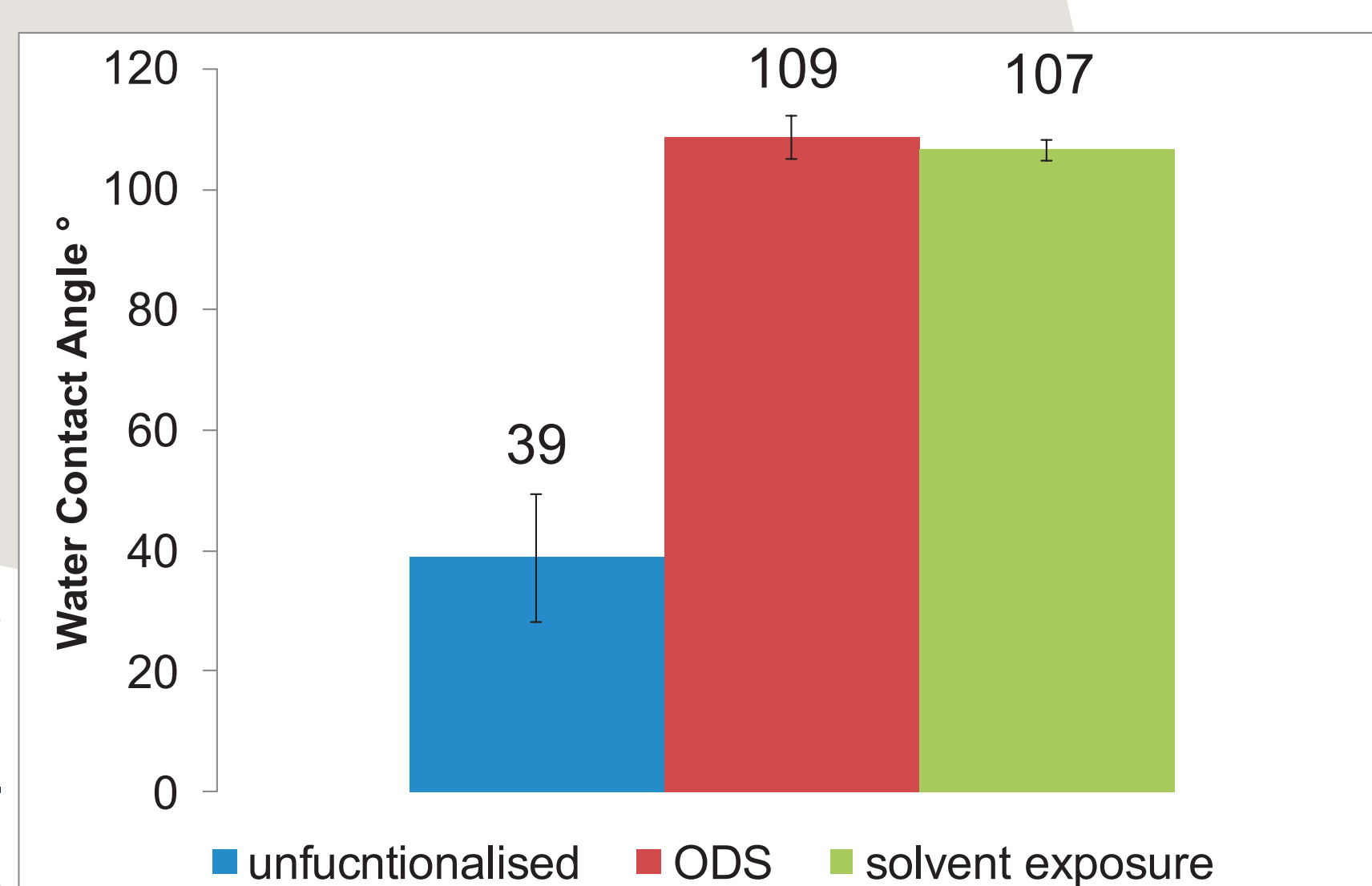


Figure 3: Optical Micrograph of cellulose beads—scale bar = 400 μm

Results

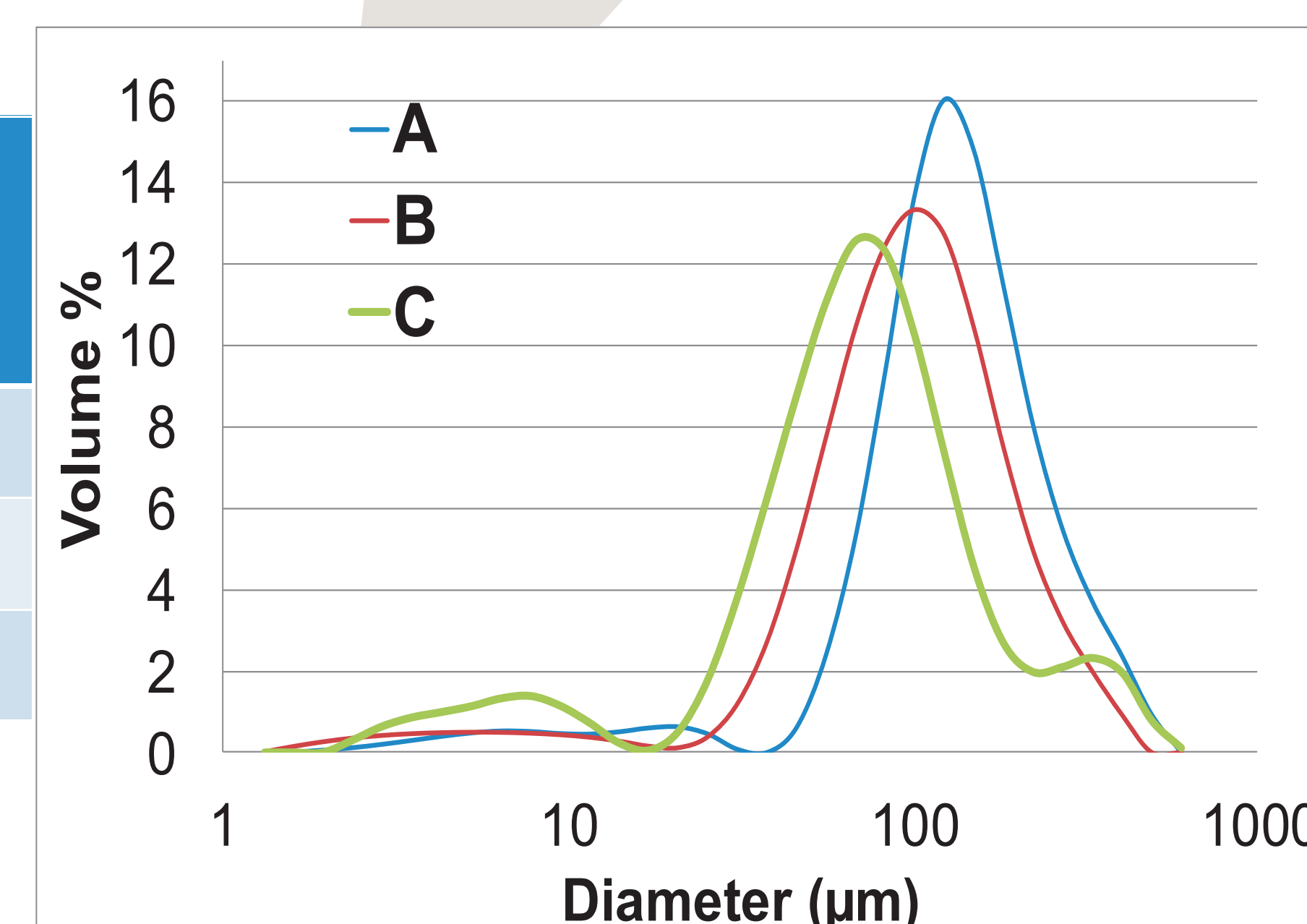
Membrane hydrophobisation

A hydrophobic membrane was required to reduce the interaction of the aqueous phase (EMIMAc, DMSO, cellulose) with the membrane. Shirasu Porous Glass (SPG) membranes were hydrophobised with 5 vol% octadecyltri-chlorosilane (ODS), a functionality that was found to be stable in the solvent system (DMSO, EMIMAc—42 hrs 80°C).



Influence of Processing Conditions

| | Cellulose wt% | Conti. Phase Flow Rate (Lmin ⁻¹) | Conti. Phase Temp (°C) | Surf. Wt% |
|---|---------------|--|------------------------|-----------|
| A | 4 | 0.4 | 30 | 0.25 |
| B | 6 | 1.4 | 45 | 1.13 |
| C | 8 | 2.4 | 60 | 2.00 |



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